



IN THE UNITED STATES PATENT  
AND TRADEMARK OFFICE

Applicant(s) : Mikihiro ENDO et al.

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For : SURFACE-PROTECTIVE PRESSURE-SENSITIVE  
ADHESIVE SHEET

Art Unit : 1771

Examiner : V. Chang

Hon. Commissioner of Patents  
and Trademarks  
Washington, D.C. 20231

DECLARATION UNDER 37 CFR 1.132

S I R :

I. Mikihiro ENDO, a citizen of Japan, who declares and says that:

I am an inventor of the present U.S. Patent Application as identified above and understand the English language. I studied the Final Office Action dated July 9, 2003 received in said application, and in order to prove that the present invention is not obvious over the references cited by the Examiner, the following experiments were carried out under my supervision.

II. Experiments

(1) Preparation of sample according to the present invention

In the same manner as in Example 3 mentioned on pages 11 and 12 of the present specification, a pressure-

sensitive adhesive sheet for surface protection was prepared and used for the following evaluation tests.

(2) Comparison with JP 07026212

Pressure-sensitive adhesive sheets described in Example 1 and Comparative example 1 of JP 07026212 were prepared with the following constitution.

(a) Example 1 of JP 07026212

	Layer	Composition	Thickness
	Polyolefin type resin surface layer	MIRASON 12	20 $\mu$ m
(a)	TiO-containing polyolefin type resin layer	MIRASON 12/MT-500HD =95/5	10 $\mu$ m
(b)	Polyolefin type resin surface layer	MIRASON 12	20 $\mu$ m
(c)	Acid-modified block copolymer layer	Tough tech M1943	5 $\mu$ m
(d)	Adhesive	BA/EA/AA=89/10/1	10 $\mu$ m

(b) Comparative example 1 of JP 07026212

	Layer	Composition	Thickness
(b)	Polyolefin type resin single layer	MIRASON 12	50 $\mu$ m
(d)	Adhesive	BA/EA/AA=89/10/1	10 $\mu$ m

(3) Evaluation of samples on weathering resistance

The above-mentioned samples were applied to a poor adherend acrylic paint film, followed by irradiation with carbon arc. The thus treated samples were then subjected to peeling at a rate of 300 mm/min to evaluate if fracture occurred or not in the substrate.

The results are shown in the following Tables 1 and 2.

Table 1  
Adhesive force (g/25 mm) to coated plate after irradiation  
by Sunshine Weather meter

Material to be adhered	Adhesive		Example 3 of present invention	Example 1 of JP 07026212	Comparative example 1 of JP 07026212
Coated plate (black)	Rubber type of present invention	Not irradiated	4.3	1.2	2.3
		After irradiation of 100 hrs	6.7	10 adhesive transfer	0.7 adhesive transfer
		After irradiation of 500 hrs	7.3	4.0 adhesive transfer	0.5 adhesive transfer
	Acryl type of JP 07026212	Not irradiated	0.7	1.8	0.8
		After irradiation of 100 hrs	1.5	3.5	1.3 adhesive transfer
		After irradiation of 500 hrs	1.8	4.2	1.7 adhesive transfer

Coated plate: 2K clear/Black metallic (which is a paint available from Standox Corp.)

\* adhesive transfer: guy anchor failure or cohesive failure

Table 2  
Adhesive force (g/25 mm) to stainless plate after  
irradiation by Sunshine Weather meter

Material to be adhered	Adhesive		Example 3 of present invention	Example 1 of JP 07026212	Comparative example 1 of JP 07026212
SP plate	Rubber type of present invention	Not irradiated	6.5	6	4.5
		After irradiation of 100 hrs	7.8	5.8 adhesive transfer	0.8 adhesive transfer
		After irradiation of 500 hrs	8.8	1.7 adhesive transfer	0.5 adhesive transfer
	Acryl type of JP 07026212	Not irradiated	0.7	1.7	0.7
		After irradiation of 100 hrs	1.8	4.8	1.5 adhesive transfer
		After irradiation of 500 hrs	2.2	9.2 adhesive transfer	1.5 adhesive transfer

When the above results are reformulated based on the evaluation method mentioned in the present specification, they are as shown below.

Table 3

Adhesiveness between substrate and adhesive layer after irradiation of 500 hours by Sunshine Weather meter

Material to be adhered	Adhesive	Example 3 of present invention	Example 1 of JP 07026212	Comparative example 1 of JP 07026212
Coated plate (black)	Rubber type of present invention	○	X	X
	Acryl type of JP 07026212	○	○	X

○: No peeling of adhesive layer, X: Adhesive layer peeled off.

Table 4

Adhesiveness between substrate and adhesive layer after irradiation of 500 hours by Sunshine Weather meter

Material to be adhered	Adhesive	Example 3 of present invention	Example 1 of JP 07026212	Comparative example 1 of JP 07026212
Stainless plate	Rubber type of present invention	○	X	X
	Acryl type of JP 07026212	○	X	X

○: No peeling of adhesive layer, X: Adhesive layer peeled off.

As can be seen from the above results, the sample of the present invention showed no adhesive transfer than that of the sample of Example 1 of JP 07026212, particularly in case of using a rubber type adhesive as an adhesive. Also, weathering resistance, particularly UV-ray cutting ability, of the sample of the present invention is excellent as

compared to that of the sample of Example 1 of JP 07026212 irrespective of the kind of the adhesive.

(4) Evaluation of samples on SUV irradiation

By using the above-prepared samples, status and color change in a coated film after irradiation by using a Super UV tester Type SUV-W151 (manufactured by Iwasaki Denki K.K.) with an irradiation of 90 mW/cm<sup>2</sup> for a setting time of 120 hours (4 hours irradiation + 4 hours rest + 4 hours dew) x 10 cycles were observed. The results are as shown in the following Tables 5 and 6.

Table 5  
Status after 120 hours SUV irradiation

Test piece	Adhesive	Example 3 of present invention	Example 1 of JP 07026212	Comparative example 1 of JP 07026212
Coated plate (white)	Rubber type of present invention	Possible to peel off	Impossible to peel off guy anchor failure substrate failure	Impossible to peel off guy anchor failure substrate failure
	Acryl type of JP 07026212	Possible to peel off	Possible to peel off	Impossible to peel off guy anchor failure substrate failure
Coated plate (black metallic)	Rubber type of present invention	Possible to peel off	Impossible to peel off guy anchor failure substrate failure	Impossible to peel off guy anchor failure substrate failure
	Acryl type of JP 07026212	Possible to peel off	Impossible to peel off guy anchor failure substrate failure	Impossible to peel off guy anchor failure substrate failure

Table 6  
Status of yellow color change ( $\Delta E$  value) of coated film after 120 hours SUV irradiation

Test piece	Adhesive	Example 3 of present invention	Example 1 of JP 07026212	Comparative example 1 of JP 07026212	No adhesion (for reference)
Coated plate (white)	Acryl type	0.9	4.1	Could not be measured due to guy anchor failure	6.5

Color difference measurement conditions:

Color Analyzer TC-1800D8 manufactured by Tokyo Denshoku K.K., reflective light, CIE 2° observer, Lab color display system, CIE standard illuminant C.

In Example 1 of JP 07026212, the adhesive remained at the time of peeling onto the black coating film. In the sample of the present invention, no adhesive remained on the coating film and the sheet can be well peeled off.

In yellow color change after SUV irradiation for 120 hours, the white coating film was less color changed when the adhesive sheet of the present invention was adhered thereon.

When the above results are reformulated based on the evaluation method mentioned in the present specification, they are as shown below.

Table 7  
Substrate failure at the time of peeling  
after 120 hours SUV irradiation

Test piece	Adhesive	Example 3 of present invention	Example 1 of JP 07026212	Comparative example 1 of JP 07026212
Coated plate (white)	Rubber type of present invention	○	○	X
	Acryl type of JP 07026212	○	○	X
Coated plate (black metallic)	Rubber type of present invention	○	X	X
	Acryl type of JP 07026212	○	X	X

○: No failure of the substrate at the time of peeling,  
X: Failure of the substrate occurred at the time of peeling.

Thus, the adhesive sheet of the present invention showed most excellent result as compared to those of the samples described in JP 07026212.

(5) Comparison with JP 11021519

A pressure-sensitive adhesive sheet described in Example 1 of JP 11021519 was prepared with the following constitution.

(a) Example 1 of JP 11021519

	Composition	Example 1 of JP 11021519
Surface layer	High density polyethylene	70
	Low density polyethylene	30
Substrate layer	Polypropylene	100
Adhesive layer	Propylene 1-butene-4-methylpentene copolymer (Replaced with propylene 1-butene copolymer)	60
	Ethylene-propylene copolymer	25
	SEPS	15

(6) Evaluation of samples on SUV irradiation

By using the above-prepared sample and the sample of the present invention (Example 3) in combination as mentioned below, status and color change in a coated film after irradiation by using a Super UV tester Type SUV-W151 (manufactured by Iwasaki Denki K.K.) with an irradiation of 90 mW/cm<sup>2</sup> for a setting time of 120 hours (4 hours irradiation + 4 hours rest + 4 hours dew) x 10 cycles were observed. The results are as shown in the following Table 8.

Table 8

	Composition	Example 1 of JP 11021519	Comparative sample 1	Comparative sample 2	Example 3 of present invention	No adhesion
Surface layer	High density polyethylene	70	70	Substrate of Example 3 of present invention	Substrate of Example 3 of present invention	
	Low density polyethylene	30	30			
Substrate layer	Polypropylene	100	100			
Adhesive layer	Propylene 1-butene-4-methylpentene copolymer (Replaced with propylene 1-butene copolymer)	60	Adhesive of Example 3 of present invention	60	Adhesive of Example 3 of present invention	
	Ethylene-propylene copolymer	25		25		
	SEPS	15		15		
Status after 120 hours SUV irradiation	Status of peeling	Difficult to peel off substrate failure	Difficult to peel off substrate failure	Possible to peel off	Possible to peel off	
	Color difference $\Delta E$ (after peeling) of coating film to which the sheet adhered	9.6	10	1.1	0.5	6.9

Conditions for accelerated exposure by S-UV and measurement conditions of color difference are the same as those mentioned above.



Thus, it would be clear that the adhesive sheet of the present invention showed most excellent result as compared to those of the sample described in JP 11021519.

### III. Conclusion

From the results shown in the above-mentioned Tables, when the results of the adhesive sheets of the present invention and those of JP 07026212 and JP 11021519 are compared to each other, it was found that the adhesive sheet of the present invention has excellent surface protective properties as compared with those of the samples shown in the reference cited by the Examiner.

Also, whereas the uses of JP 07026212 and JP 11021519 are surface protection alone for common use, but that of the present invention is to protect a coated film for an automobile which is required to have excellent weathering property, so that they are quite different in final objects from each other. The substrates used in Example 1 of JP 07026212 and Example 1 of JP 11021519 are both transparent, and protection properties (shielding effect) for the coated film to light of these protective films are inferior to that of the present invention as shown in the above-mentioned tables, so that these films are considered to be inappropriate for the use of protection for coated films of an automobile required in high weathering property. In particular, a transparent protective film which is one of the characteristic feature of JP 07026212 is considered to be inappropriate for the use of protection for coated films of an automobile.

Such effects of the present invention would indeed be remarkable and could not be expected from the description of the cited references. Thus, I do not believe that the present invention can be easily expected from the

descriptions of the references.

IV. I further declare that all statements made herein of my own knowledge are true and that all statements made in information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001, of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated: 09.01.2004

Mikihiro Endo

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